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TITLE: Manufacture of isoflavone compound
from soybean germ - involves removing soybean germ using
water-soluble organic solvent and lipophilic
components using hydrophobic organic solvent followed
by refining and drying the extract

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PATENT-FAMILY:

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INT-CL (IPC): A61K031/35, A61K035/78 , C07D311/36

ABSTRACTED-PUB-NO: JP 11263786A

BASIC-ABSTRACT:

NOVELTY - Soybean germ is removed by water-soluble organic solvent and lipophilic components are removed by hydrophobic organic solvent, after extraction concentration. The extract is finally refined and dried to obtain an isoflavone compound.

USE - The method is useful for manufacturing isoflavone compounds, from soybean germ obtained from soybean protein or natto.

ADVANTAGE - Isoflavone can be manufactured economically in large quantities.

CHOSEN-DRAWING: Dwg.1/1

TITLE-TERMS: MANUFACTURE COMPOUND SOY GERM REMOVE SOY GERM WATER SOLUBLE

ORGANIC SOLVENT LIPOPHILIC COMPONENT
HYDROPHOBIC ORGANIC SOLVENT
FOLLOW REFINE DRY EXTRACT

DERWENT-CLASS: B02

CPI-CODES: B06-A01;

CHEMICAL-CODES:

Chemical Indexing M2 *01*

Fragmentation Code

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H403 H441 H442 H443 H541 H542 H8 J5 J521 M1
M113 M210 M211 M272 M280 M281 M282 M320 M412 M511
M520 M531 M540 M720 M903 M904 N161

Markush Compounds

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(54)【発明の名称】 イソフラボン化合物の製造法

(57)【要約】

【課題】 大豆胚芽を原料として安価で大量にイソフラボン化合物を精製・製造することを課題とする。

【解決手段】 大豆胚芽を原料として、抽出液の吸着樹脂処理に先だって、疎水性有機溶媒洗滌または遠心分離により精製効果を高めることが有効であることを見出した。

【特許請求の範囲】

【請求項1】 大豆胚芽を水溶性有機溶媒により抽出濃縮後、疎水性有機溶媒により親油性成分を除去し、再び濃縮、精製及び乾燥することを特徴とするイソフラボン化合物の製造法。

【請求項2】 大豆胚芽を水溶性有機溶媒により抽出濃縮後、生ずる乳化固形物を遠心分離により除去し、その後精製及び乾燥することを特徴とするイソフラボン化合物の製造法。

【発明の詳細な説明】

*【0001】

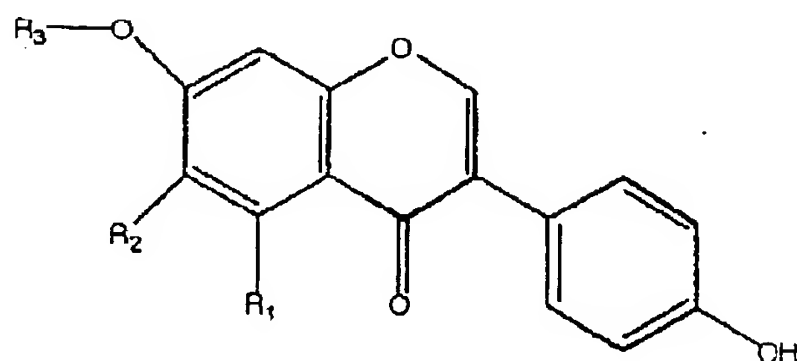
【発明の属する技術分野】本発明は、イソフラボン化合物の製造法に関する。

【0002】

【従来の技術】マメ科 (Leguminosae) の植物であるダイズの種子、大豆は貴重な植物蛋白源として古くより栽培されており、種々の食品の原料となっている。この大豆には、蛋白質、糖質、ビタミン類の他に、下記式

【0003】

*10 【化1】



【0004】

※20※【表1】

化合物名	R ₁	R ₂	R ₃
ダイズイン	H	H	グルコース
グリシチン	H	OCH ₃	グルコース
ゲニスチン	OH	H	グルコース
ダイゼイン	H	H	H
グリシテイン	H	OCH ₃	H
ゲニステイン	OH	H	H

【0005】で表されるダイズイン、グリシチン、ゲニスチン、ダイゼイン、グリシテイン、ゲニステイン等のイソフラボン化合物が含まれており、最近では、閉経前の女性の女性ホルモンに対するイソフラボンの影響を調べ、その影響を確認したり、(Br. J. of Nutrition Vol.74, p587-601(1995))、ゲニステインがチロシンキナーゼ阻害剤であり、アジア人の大豆摂取は前立腺がん、乳がんを予防するという疫学的調査に基づいて、臨床試験が行なわれたり(J. Cell Biochemistry No.22 p181-187(1995))、また、イソフラボンはカルシウムの体内から流出を防ぐのに効果があり、更年期における骨そしょう症等に有効であるとされており、その医薬品あるいは健康補助食品としての用途が注目されている。

【0006】従つて、今後これらのイソフラボン化合物を医薬品あるいは健康補助食品として提供する場合、いかに安価に、かつ大量にイソフラボン化合物を得るかが重要な問題となる。従来、イソフラボン化合物を得るには、大豆、大豆抽出液である煮汁、醤油粕、醤油油等を原料として用い、有機溶媒または、含水有機溶媒による抽出液をアルミナ、シリカゲル、合成吸着樹脂等を吸着

★剤とするカラムクロマトグラフィーで精製する方法がとられていた。

【0007】しかし、これらの方法を工業的に大量精製に適用する場合、原料のイソフラボン含量が低いことから、廃棄物が多量に発生し、精製の為の操作が煩雑になる等の不都合が生じるため、得られるイソフラボン化合物は少量でしかも高価なものになりイソフラボン化合物の工業的な精製はほとんど行なわれていなかった。大豆胚芽には比較的多くのイソフラボンが含まれていることは知られているが、同時に親油性成分を多量に含み、そのため単なる水溶性有機溶媒抽出物の濃縮物では多量の乳化固形物を生じ、これが無極性の多孔性合成吸着樹脂による精製を著しく妨害する要因となっていた。

【0008】

【発明が解決しようとする課題】そこで本発明は、大豆胚芽を原料として安価で大量にイソフラボン化合物を精製・製造することを課題とする。

【0009】

【課題を解決するための手段】本発明者は、上記課題を解決すべく鋭意努力した結果、大豆胚芽を原料として、

抽出液の吸着樹脂処理に先だって、疎水性有機溶媒洗滌または遠心分離により精製効果を高めることが有効であることを見出した。

【0010】すなわち、本発明は、(1)大豆胚芽を水溶性有機溶媒により抽出濃縮後、疎水性有機溶媒により親油性成分を除去し、再び濃縮、精製及び乾燥することを特徴とするイソフラボン化合物の製造法、(2)大豆胚芽を水溶性有機溶媒により抽出濃縮後、生ずる乳化固形物を遠心分離により除去し、その後精製及び乾燥することを特徴とするイソフラボン化合物の製造法に関する。

【0011】本発明のイソフラボン化合物の製造方法は、まずダイズ蛋白、納豆等の製造工程で得られる大豆胚芽を出発原料として、これを水溶性有機溶媒または含水有機溶媒で抽出、濃縮、疎水性有機溶媒を用いて液液分配により、親油性成分を疎水性有機溶媒層に除去し、再び水溶液層を濃縮、有機溶媒を留去し、該当する合成吸着樹脂に通し、イソフラボン化合物を吸着させ、次いで水または低含量の有機溶媒を加えた水で不純物を洗浄し、有機溶媒または有機溶媒と水との混合溶媒を用いて

該当する合成樹脂から溶出させ、濃縮、乾燥することを特徴とするものである。

【0012】また、疎水性有機溶媒を用いた液液分配に代えて、有機溶媒を留去するまで濃縮し、生じた乳化固形物を遠心分離により除去後、該当する合成吸着樹脂に通し、前記の方法によりイソフラボン化合物を製造する方法も本発明の特徴である。

【0013】本発明において、該当する合成吸着樹脂の具体例としては、「ダイヤイオンHP樹脂」(三菱化学社製)、「アンバーライトXAD樹脂」(ロームアンドハース社製)、「デュオライトS樹脂」(ダイヤモンドシャムロック社製)等が挙げられる。なお、本発明の製造フロー例を、図1に示す。

【0014】以下に本発明を詳細に説明する。大豆(Glycine max Merrill)由来の胚芽はダイズ蛋白の製造工程で得られるものを利用することができ、この大豆胚芽を抽出溶媒で抽出する。抽出溶媒は、メタノール、エタノール、アセトンなどの水溶性有機溶媒または含水有機溶媒を用いることができ、室温から抽出溶媒の沸点までの温度範囲内で抽出できる。また、70℃以上の熱水を用いても抽出できる。濃縮は、減圧下、または、常圧下で行い、室温から抽出溶媒の沸点までの温度範囲内で行う。

【0015】精製度の高いイソフラボン化合物を得るため、上記記載大豆胚芽抽出液を濃縮し、疎水性有機溶媒を用いて液液分配を行う。疎水性有機溶媒としてはヘキサン、酢酸エチル、エーテル等を用いることができる。液液分配後、水溶液層を濃縮、有機溶媒を留去し、該当する合成吸着樹脂にイソフラボン化合物を吸着させ、次いで水または低含量の有機溶媒を加えた水で不純物を洗浄し、有機溶媒または有機溶媒と水との混合溶媒を用い

て該当する合成樹脂から溶出させ、濃縮、乾燥し、抽出固形物を得る。また、大豆抽出液を濃縮し、生成する乳化固形物を遠心分離により除去し、該当する合成吸着樹脂にイソフラボン化合物を吸着させ、次いで水または低含量の有機溶媒を加えた水で不純物を洗浄し、有機溶媒または有機溶媒と水との混合溶媒を用いて該当する合成樹脂から溶出させ、濃縮、乾燥し、抽出固形物を得る。

【0016】イソフラボン化合物吸着は、通常、カラム法で行われ、室温下で行う。洗浄に有機溶媒を用いる場合は、ダイヤイオンHP20の場合、メタノール、エタノール、アセトンが望ましく、その含量は20%以下が望ましい。ダイヤイオンHP20の場合、溶出に用いる有機溶媒はメタノール、エタノール、アセトンが望ましく、これら有機溶媒を含水で用いる場合は、水分を70%以下とするのが望ましい。濃縮、乾燥は、大豆胚芽からの有機溶媒抽出固形物の場合と同様にして行う。本発明のイソフラボン化合物の製造法で使用した該当する合成吸着樹脂は、適当な有機溶媒(例えば、アルコール系、アセトン系有機溶媒等)または、アルカリ剤(例えば、水酸化ナトリウム、水酸化カリウム等)により洗浄再生することにより、繰り返し使用が可能である。

【0017】

【発明の実施の形態】次に実施例を示して本発明を更に詳しく説明するが、本発明はこれにより何ら制限されるものではない。

【実施例1】大豆蛋白製造工程で得られる大豆胚芽1kgを90%メタノール3000mlで60℃、1.0時間の加温抽出を4回繰り返し抽出を行った。この抽出液を50~60%になるよう溶媒を減圧濃縮し、濃縮液をヘキサン500mlで分配し、下層液を更に減圧濃縮して十分にメタノールを留去し、固形分10%になるまで濃縮した。

【0018】活性化させたスチレンジビニルベンゼン重合樹脂(三菱化成工業株式会社製、ダイヤイオンHP20)を1000mlを充填した樹脂塔に添加し、樹脂塔内を1000ml/hrで通過させ、次いで2000mlの水で洗浄した。洗浄後、樹脂塔にメタノールを3000ml通液させ、イソフラボン化合物を含有する溶液を得た。この溶液は、減圧下で60℃で濃縮、噴霧乾燥し、イソフラボン化合物をアグリコンにして10.0%含む抽出固形物を54.7gを得た。比較例1に比較してイソフラボン化合物含量は17.6%、イソフラボン化合物収率は56.2%増大した。

【0019】【実施例2】大豆蛋白製造工程で得られる大豆胚芽100gを80℃、熱水1000mlで1.0時間、700mlで2回抽出し、減圧下60℃で約300mlまで濃縮後、濃縮後生成した乳化固形分を4200gの遠心分離にて除去した後、活性化させたスチレンジビニルベンゼン重合樹脂(三菱化成工業株式会社製、ダイヤイオンHP20)を200mlを充填した樹脂塔に添加し、樹脂塔内を200ml/hrで通過させ、次いで400mlの水で洗浄した。洗浄後、樹脂塔に600mlメタノールを通過させ、イソフラボン化合物を含有する溶

液を得た。

【0020】この溶液は、減圧下で60℃で濃縮、噴霧乾燥し、イソフラボン化合物をアグリコンにして11.2%含む抽出固形物を3.8g得た。比較例1に比較して、イソフラボン化合物含量は31.8%、イソフラボン化合物収率は21.5%増大した。

【0021】[比較例1]大豆蛋白製造工程で得られる大豆胚芽1kgを90%メタノール3000mlで60℃、1.0時間の加温抽出を4回繰り返し抽出を行った。減圧濃縮して十分にメタノールを留去し、固形分10%になるよう濃縮した。この濃縮液は非常に多くの沈殿物を生じたが、これをこのまま、活性化させたスチレンジビニルベンゼン重合樹脂(三菱化成工業株式会社製、ダイヤイオンHP20)1000mlを充填した樹脂塔に添加し、樹脂塔内を1000ml/hrで通過させた。この場合、通液中にカラム上層に沈殿物が堆積し通液速度の減少を認めた。

【0022】次いで2000mlの水で洗浄したが、十分な通液速度を得ることができなかった。樹脂塔に3000mlメタノールを通過させ、イソフラボン化合物を溶出させた。

この溶出液を減圧下で60℃で濃縮、噴霧乾燥し、イソフラボン化合物をアグリコンにして8.5%含む抽出固形物を41.2gを得た。イソフラボンの定量は、高速液体クロマトグラフィーを用いており、サンプルを5%塩酸溶液1容、メタノール1容の溶媒に溶解し、温浴中で3時間加熱還流して加水分解し、アグリコンをODSカラムを用い、検出波長260nmにて測定した。標品としてダイゼイン、ゲニステインを用いて定量した。このカラム処理に影響した沈殿物について薄層クロマトグラフィーと各種発色試薬で解析したが、乳化作用を示すリン脂質を確認した。

【0023】

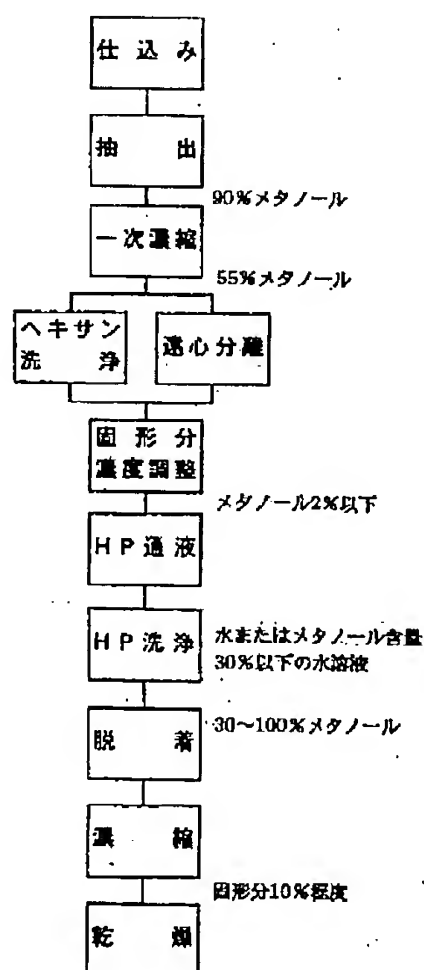
【発明の効果】本発明により、イソフラボン化合物を安価に大量に製造することができ、医薬的效果を有効に利用することが可能となる。

【図面の簡単な説明】

【図1】イソフラボン高含量濃縮固形物製造フローを示す図。

【図1】

イソフラボン高含量濃縮固形物製造フロー



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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacturing method of an isoflavone compound.

[0002]

[Description of the Prior Art] The seed of the soybeans which are the vegetation of Leguminosae (Leguminosae), and soybean are grown more in ancient times as a precious source of vegetable albumin, get down, and serve as a raw material of various food. In this soybean, it is [others / protein, sugar, and vitamins] the following type [0003].

[Formula 1]

[0004]

[Table 1]

[0005] It comes out and isoflavone compounds, such as the soybean in expressed, glycitin, genistin, a die zein, glycite and genistein, are contained. Recently The effect of isoflavone to the female sex hormone of the woman in front of a menopause is investigated. Check the effect or (Br.J.of Nutrition Vol.74, p587-601 (1995)), Genistein is a tyrosine kinase inhibitor. Soybean intake of the people from Asia A prostatic cancer, It is based on the epidemiological survey

preventing a breast cancer. A clinical trial is performed or (J.Cell Biochemistry No.22 p181-187 (1995)), Moreover, in isoflavone protecting an outflow from the inside of the body of calcium about effectiveness, it is supposed that it is effective in ***** in the menopause etc., and the application as the drugs or a health supplement attracts attention.

[0006] Therefore, when these isoflavone compounds will be offered as drugs or a health supplement from now on, how an isoflavone compound is obtained cheaply and in large quantities poses an important problem. In order to have obtained the isoflavone compound conventionally, the approach of refining by the column chromatography which uses an alumina, silica gel, synthetic adsorption resin, etc. as an adsorbent was taken in the extract by the organic solvent or the water organic solvent, using an soybean, the stock which is an soybean extract, soy meal, soy oil, etc. as a raw material.

[0007] However, since the isoflavone content of a raw material was low when applying these approaches to extensive purification industrially, trash was generated so much, since un-arranging -- the actuation for purification becomes complicated -- arose, it became little [the isoflavone compound obtained] and expensive moreover, and most industrial purification of an isoflavone compound was not performed. Although it was known that comparatively much isoflavone is contained in an soybean germ, for the reason, including an oleophilic component so much, by the concentrate of a water-soluble mere organic solvent extract, a lot of emulsification solids were produced in coincidence, and this had become the factor which blocks remarkably purification by non-polar porous composition adsorption resin at it.

[0008]

[Problem(s) to be Solved by the Invention] Then, this invention makes it a technical problem to be cheap as a raw material, and to refine and manufacture an isoflavone compound for an soybean germ in large quantities.

[0009]

[Means for Solving the Problem] this invention person found out that it was effective to heighten the purification effectiveness according to hydrophobic organic solvent washing or centrifugal separation in advance of the adsorption resin treatment of an extract by using an soybean germ as a raw material, as a result of trying hard wholeheartedly that the above-mentioned technical problem should be solved.

[0010] That is, a water-soluble organic solvent removes (1) soybean germ, it removes an oleophilic component with a hydrophobic organic solvent after extract concentration, and this invention removes the emulsification solid which produces the manufacturing method of the isoflavone compound characterized by condensing, refining and drying again and (2) soybean germ after extract concentration with a water-soluble organic solvent according to centrifugal separation, and relates to the manufacturing method of the isoflavone compound characterized by refining and drying after that.

[0011] The manufacture approach of the isoflavone compound of this invention uses as a start raw material the soybean germ first obtained by production processes, such as soybean protein and fermented soybeans, and an extract, concentration, and a hydrophobic organic solvent are used for this with a water-soluble organic solvent or a water organic solvent. With liquid-liquid distribution Remove an oleophilic component in a hydrophobic organic solvent layer, condense a water-solution layer again, and an organic solvent is distilled off. Through and an isoflavone compound are made to stick to the corresponding synthetic adsorption resin, the water which subsequently added water or the organic solvent of a low content washes an impurity, elution is carried out from the synthetic resin which corresponds using the mixed solvent of an organic solvent or an organic solvent, and water, and it is characterized by condensing and drying

[0012] Moreover, the method of manufacturing an isoflavone compound by through and the aforementioned approach to the synthetic adsorption resin which corresponds after removal the emulsification solid which replaced with the liquid-liquid distribution using a hydrophobic organic solvent, condensed until it distilled off the organic solvent, and was produced according to centrifugal separation is also the description of this invention.

[0013] In this invention, "diamond ion HP resin" (Mitsubishi Chemical make), "Amberlite MB3 mixed bed ion exchange resin" (Rohm and Haas Company make), "Duolite S resin" (diamond SHAMU lock company make), etc. are mentioned as an example of the corresponding synthetic adsorption resin. In addition, the example of a manufacture flow of this invention is shown in drawing 1 <http://www4.ipdl.ncipi.go.jp/cgi-bin/tran_web.cgi_ejje?u=http%3A%2F%2Fwww4.ipdl.ncipi.go.jp%2FTokujitu%2Ftjitemdrw.ipdl%3FN0000%3D237%26N0500%3D1E%5FN%2F%3B%3E

[0014] This invention is explained below at a detail. The germ of the soybean (*Glycine max* Merrill) origin can use which is obtained by the production process of soybean protein, and extracts this soybean germ with an extracting solvent. A water-soluble organic solvent or water organic solvents, such as a methanol, ethanol, and an acetone, can be used for extracting solvent, and it can extract them from a room temperature in the temperature requirement by the boiling point of an extracting solvent. Moreover, even if it uses hot water 70 degrees C or more, it can extract. Concentration is performed under reduced pressure or ordinary pressure, and it carries out in the temperature requirement from a room temperature to the boiling point of an extracting solvent.

[0015] In order to obtain the high isoflavone compound of whenever [purification], the above-mentioned written soybean germ extract is condensed, and liquid-liquid distribution is performed using a hydrophobic organic solvent. A hexane, ethyl acetate, the ether, etc. can be used as a hydrophobic organic solvent. Condense a water-solution layer after liquid-liquid distribution, and distill off an organic solvent, an isoflavone compound is made to stick to the corresponding synthetic adsorption resin, the water which subsequently added water or the organic solvent of a low content washes impurity, elution is carried out from the synthetic resin which corresponds using the mixed solvent of an organic solvent or an organic solvent, and water, it condenses and dries and an extract solid is obtained. Moreover, an soybean extract condensed, centrifugal separation removes the emulsification solid to generate, an isoflavone compound is made to stick to the corresponding synthetic adsorption resin, the water which subsequently added water or the organic solvent of a low content washes an impurity, elution is carried out from the synthetic resin which corresponds using the mixed solvent of an organic solvent or an organic solvent, and water, it condenses and dries and an extract solid is obtained.

[0016] Isoflavone compound adsorption is performed with a column method, and is usually performed under a room temperature. When using an organic solvent for washing, in the case of the diamond ion HP20, a methanol, ethanol, and an acetone are desirable and 20% or less of the content is desirable. A methanol, ethanol, and its acetone are desirable and when using these organic solvents by water, as for the organic solvent which is used for elution in the case of the diamond ion HP20, it is desirable to make moisture into 70% or less. Concentration and desiccation are performed like the case of the organic solvent extract solid from an soybean germ. Repeat use is possible for the corresponding synthetic adsorption resin which was used by the manufacturing method of the isoflavone compound of this invention carrying out washing playback by suitable organic solvents (for example, an alcoholic system, an acetone system organic solvent, etc.) or alkali chemicals (for example, a sodium hydroxide, a potassium hydroxide, etc.).

[0017]

[Embodiment of the Invention] Next, although an example is shown and this invention is explained in more detail, thereby, this invention is not restricted at all.

1kg of soybean germs obtained by the [example 1] soybean-protein production process -- 90% methanol 3000ml -- warming of 60 degrees C and 1.0 hours -- the repeat extract was extracted 4 times. Vacuum concentration of the solvent was carried out so that it might become 50 - 60% about this extract, concentration liquid was distributed by hexane 500ml, vacuum concentration of the lower layer liquid was carried out further, and the methanol was fully distilled off and it condensed until it became 10% of solid content.

[0018] activated styrene divinylbenzene polymerization resin (the Mitsubishi Kasei Industries make, diamond ion HP20) the resin filled up with 1000ml -- a column -- adding -- resin -- a column -- inside was passed by 1000 ml/hr and, subsequently 2000ml water washed. after washing and resin -- the column was made to dip 3000ml of methanol, and the solution containing an isoflavone compound was obtained. This solution obtained 54.7g for concentration and the extract solid which carries out spray drying, makes an isoflavone compound an aglycon and contains it 10.0% at 60 degrees C under reduced pressure. In the isoflavone compound content, as compared with the example 1 of a comparison, isoflavone compound yield increased 56.2% 17.6%.

[0019] 100g of soybean germs obtained by the [example 2] soybean-protein production process 80 degrees C, 1000ml hot water extracts twice by 700ml for 1.0 hours. To about 300ml at 60 degrees C under reduced pressure After concentration, After removing the emulsification solid content generated after concentration in 4200g centrifugal separation, activated styrene divinylbenzene polymerization resin (the Mitsubishi Kasei Industries make, diamond ion HP20) the resin filled up with 200ml -- a column -- adding -- resin -- a column -- inside was passed by 200 ml/hr and, subsequently 400ml water washed. after washing and resin -- the column was made to pass 600ml methanol and the

solution containing an isoflavone compound was obtained.

[0020] This solution obtained concentration and 3.8g of extract solids which carry out spray drying, make an isoflavone compound an aglycon and contain it 11.2% at 60 degrees C under reduced pressure. In the isoflavone compound content, as compared with the example 1 of a comparison, isoflavone compound yield increased 21.5% 31.8%.

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[0022] Subsequently, although 2000ml water washed, sufficient dipping rate was not able to be obtained. resin -- the column was made to pass 3000ml methanol and elution of the isoflavone compound was carried out. 41.2g was obtained for concentration and the extract solid which carries out spray drying, makes an isoflavone compound an aglycon and contains it 8.5% at 60 degrees C under reduced pressure of this eluate. The quantum of isoflavone dissolved the sample in the solvent of solution-of-hydrochloric-acid 1 ** and methanol 1 ** 5%, was under hot bath, high performance chromatography is used, and it carried out heating reflux for 3 hours, hydrolyzed, and measured the aglycon on the detection wavelength of 260nm using the ODS column. The quantum was carried out using a die zein and genistein as preparation. Although analyzed with thin-layer chromatography and various color reagents about the precipitate which influenced this column processing, the phospholipid which shows an emulsification operation was checked.

[0023]

[Effect of the Invention] An isoflavone compound can be manufactured in large quantities cheaply, and this invention enables it to use physic-effectiveness effectively.

[Translation done.]

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

MEANS

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